A Prototype Semi-Automated System for Measuring Air Infiltration in Buildings Using Sulfur Hexafluoride as a Tracer

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ABSTRACT

A system is described which automatically operates a small gas chromatograph and measures parts per billion concentrations of sulfur hexafluoride (SF₆) in air. It samples air on a 10-minute cycle and records the response on a strip chart recorder. When SF₆ is distributed in the air of a building, data is obtained from which air infiltration rates may be determined.

Key Words: Air infiltration instrumentation; air infiltration measurement; building ventilation rates; sulfur hexafluoride tracer

1. Introduction

Air infiltration is one of the paths of energy loss which must be assessed in analyzing and improving the energy efficiency of buildings. Experimentally it can be measured by the tracer dilution method in which a known amount of tracer gas is mixed with the building air, and the rate of decrease in concentration monitored. A number of tracer gases have been used for the purpose, but the present note concerns itself with the use of sulfur hexafluoride (SF_6) . Since the development of electron capture detectors by Lovelock [1, 2] and the observation that sulfur hexafluoride can be measured in nanogram quantities by electron capture detectors [3], this gas has come into use as a tracer of air movements [4, 5, 6] and more recently in the study of building ventilation [7, 8, 9].

Besides the high sensitivity with which the gas can be measured, it is stable, inert, and nonflammable. It is not a normal background constituent of air. One of the principle uses of SF_6 is as a dielectric in transformers. The gas is nontoxic in the concentrations used in tracer measurements and not highly toxic even when present as a major constituent in air.

There are certain problems which must be dealt with in using SF_6 as a tracer. For example, the detector may require rather frequent calibration because of drift. Also because of the high sensitivity of the measurement, trace leaks in regulators, valves and connections which would go unnoticed in other gas measurement systems are completely unacceptable when SF_6 is used as an indoor tracer. In order to obtain high sensitivity a chromatograph is used. This means that measurements are in the form of chromatographic peaks which may require a little more work to automate and process than a continuous output. SF_6 is not normally present in air. However, the detector responds to other halogenated compounds such as certain refrigerants and also propellants used in aerosol spray cans. These compounds occasionally appear in the indoor background. They can usually be recognized and sometimes separated, but they are a potential source of interference. Finally, electron capture detectors contain radio-active sources and require a Nuclear Regulatory Commission license to own and operate.

This Technical Note describes the use of SF_6 with a gas chromatograph to measure air infiltration in buildings, and describes a developmental automated system of operation. Some of the problems and potential sources of error in the method are described.

2. Measurement of Air Infiltration

In the measurement of air infiltration by the tracer dilution method, a tracer gas is distributed in the air of a building, and the decay in concentration measured as a function of time. The theory of the method may be outlined briefly by considering the equation:

$$\frac{dC_i}{dt} = (C_o - C_i) \frac{v}{v}$$
(1)

where C_0 and C_1 are respectively the concentrations of tracer inside and outside the building at time t. \forall is the rate at which air enters the building. It is also the rate at which air leaves the building unless there is a buildup or loss of pressure. V is the ventilated volume of the building, and v/V is the air infiltration rate expressed in air changes per unit time.

If the outside concentration of tracer is small enough to be neglected, equation 1 may be reduced to:

$$\frac{dC_{i}}{dt} = -C_{i} \frac{v}{v}$$
(2)

Under conditions of perfect mixing, equation 2 may be integrated to give:

$$\ln \frac{C_i}{C_{io}} = -\frac{v}{v}t$$
(3)

or

$$v/v = -\frac{1}{t} \ln (C_i/C_{io})$$
 (3a)

where C_{10} is the initial indoor concentration of tracer. Equations 2, 3, and 3a have the form of the radioactive decay law or the equation of the well mixed tank. Equations 3 and 3a can be solved directly, but when manual calculation is performed it is common practice to plot $\ln C_1/C_{10}$ against t and calculate the infiltration rate from the slope of the line. It is also to be noted that it is not necessary to know absolute tracer concentrations to calculate infiltration rate provided relative concentrations, C_1/C_{10} , can be determined.

3. Description of the Chromatograph

The instrument used in these measurements was a Panatek Leak Detector 2000* operated as a gas chromatograph. A picture of the device is shown in Figure 1. In operation, argon carrier gas is passed continuously through an aluminum oxide column in the apparatus and into a pulsed mode electron capture detector. Air samples are introduced into the column by means of a sample injection valve with a 0.5 ml sampling loop. Oxygen which is also an electron capturing gas is separated from SF₆ by the aluminum oxide column and passes through the detector first. SF₆ elutes next and is measured separately. Other normal constituents of air do not interfere.

[¬] Certain commercial materials and instruments are identified in this paper in order to specify experimental conditions adequately.⁻ Such identification does not imply recommendations or indorsement by the National Bureau of Standards, nor does it imply that the equipment is necessarily the best for the purpose. The Panatek 2000 leak detector is no longer produced, but a similar unit, the SF₆ B Detector, is supplied by Ion Track Instruments.

 SF_6 concentrations have been measured by simple visual observation of needle deflections of the meter. However, the present note is devoted primarily to the description of an automated system using a strip chart recorder.

4. Automation of Measurements

a. Sampling and Measurement System

An automated system for operating the chromatograph is shown in Figure 2A. A block diagram and wiring diagram are shown in Figures 2B and 2C. The system actuates sampling at selected time intervals and records the responses on a strip chart recorder. It also selects the sampling site so that air may be drawn from more than one location in a timed sequence.

Detailed operation of the device may be described by reference to Figures 2B and 2C. Timer (1) is the main control of the system. It is a 2-gang timer with a 30-second cycle. One gang operates stepping switch (2) or stepper as it is sometimes called. The other gang actuates solenoid (12) which operates the sample injection value of the chromatograph (13).

Stepper (2) selects the functions in sequence which are to be performed. It is a 40step switch and is operated in two 20-step cycles. When actuated every 30 seconds it completes a full cycle every ten minutes.

Solenoid valves (3, 4, and 5), when actuated by stepping switch (2), select the sites from which air samples are drawn. Solenoid valve (6) opens when the automatic system for introducing tracer into a building is actuated. It allows the sampling pump to draw air during tracer-feed steps. Otherwise, the pump would be pulling against a closed system, since all of the other solenoid valves are closed. If it is used to sample tracer-free air it also provides a check on leaks in the system.

Relay (7) is normally closed. It controls the operation of auxiliary sampling pump (9). It also must be closed during sampling steps for sample injection solenoid (12) to operate. During non-sampling steps the pump and the solenoid are deactivated. Relay (8) performs two functions. One pole closes during non-sampling steps and shorts out the output from the chromatograph to establish the zero-current baseline. The other pole opens during non-sampling steps and stops chart movement.

Small modifications have been made in the system since it was originally designed. For example, stepping switch (2) and sample injection solenoid (12) are operated with rectified current as shown in Figure 2. This permits use of lower voltage and reduces electrical noise. Other modifications include padding of the plunger of solenoid (12) to reduce mechanical impact and introduction of screens in the sampling lines to keep large particles of dirt out of the sampling solenoids and the pump. Also, a 2-port atomizer bulb with check valves was placed in the output of pump (9), because it was found that otherwise samples were contaminated with ambient air at the pump output during the return stroke of the pump.

b. Tracer Injection System

An automated system for introducing SF_6 into a building is shown in a block diagram in Figure 3A. A wiring diagram of the timing control of the system is shown in Figure 3B, and the relay values and pumps which deliver the tracer are shown in Figures 3C and 3D.

The operation of the system may be described by referring to Figures 3A, B, C, and D. Stepper (15) is actuated once every 10 minutes by stepper (2). The 40 steps are apportioned so that a feed cycle is initiated every 60 or 70 minutes.

Timer (16) is actuated for 30 seconds by stepper (15) at the beginning of each feed cycle. It is a 2-gang timer, one gang of which is connected so as to take over from the stepper and operate for one 10-minute cycle. The other gang controls the time of feed. By adjustment of the cam and the electrical connections it is possible to select feed times of a few seconds up to nearly 10 minutes. Manual override switch (17) is also provided to permit feed of SF_6 independently of the automated system. Air is drawn through the feed lines at all times by one or the other of two 2 liter/minute diaphragm pumps, (20) and (22). During a feed cycle tracer from a controlled leak is diluted with air and directed into the ventilating system of the building by automatically opening solenoid valve (21) and turning on pump (22). During most of the time, however, this valve and pump are off, and air is drawn through the lines and exhausted to the outside by means of solenoid (19) and pump (20). This prevents leakage of residual tracer from the line into the building when the feed is off.

Relay (18) is actuated by timer (16) and alternately operates the feed or the exhaust.

5. Instrument Calibration

It is necessary to calibrate the instrument against known concentrations of SF_6 in air. Calibration mixtures are prepared by leaking SF_6 at a measured rate into a wind tunnel operating at a known flow rate. The gas is stored in a compressed-air tank for use as needed. Samples stored this way for a month usually agree within experimental error with freshly prepared samples drawn directly from the wind tunnel. For preparing concentrations of less than 10 ppb, dilutions of the primary standard are made with a dilution system consisting of a 2liter graduated closed cylinder with a piston and stopcocks.

A convenient form of controlled leak for SF_6 can be made from a capillary tube packed with a finely ground pigment. The rate of flow of the leak is determined with a soap bubble meter [10].

For automated operation it would be desirable to eliminate the need for pre-prepared calibration mixtures. One experimental approach has been to leak SF_6 at a constant rate into a small centrifugal fan also operating at a constant rate and use the output of the fan as a source of reference gas. The system is illustrated in Figures 3D and 4. In Figure 3D a controlled standard leak <0.01 ml/min is shown. Tracer gas from this leak is passed into a small 20-cfm (566 liters/min) centrifugal fan as shown in Figure 4. From there it goes into a length of 2-inch (5.1 cm) duct and out through an orifice plate. The latter permits estimation of the air flow rate.

6. Instrument Response

Experimental plots of instrument response as a function of SF_6 concentration are presented in Figure 5. It may be seen that the response is different after several hours of warm-up than it is after the first hour or two. During the first two hours of operation the response may change measurably within 20 or 30 minutes. After several hours it may change only slightly in an hour of operation but never reaches an absolutely constant level. It is also to be noted that the response observed by reading the meter is different from the response read from the chart recorder. The reasons for this difference has not been established, but it may possibly represent differences in frictional and inertial effects in the two systems. However, in either case, over the concentration range of about 5 to 20 ppb, the response may be expressed by an empirical relationship of the form

$$C = -a \ln \frac{I}{I_o} + \lambda$$
(4)

where I is the current flow in the detector corresponding to a concentration C, I_0 is the standing current with no tracer in the detector, and a and λ are parameters determined by calibration. When $\lambda = 0$ equation 4 can be reduced to Beers' law.

Five calibrations, each consisting of five or six points such as those shown in Figure 5, were performed on different days. The following least squares equations and standard deviations were obtained: Based on Meter Readings

1a.	$C = -27.1 \ln I/I_{o}$	$\sigma = 0.5 \text{ ppb}$
2a.	$C = -22.3 \ln I/I_{o} + 0.6$	0.3
3a.	$C = -29.3 \ln I/I_{o} - 0.4$	0.5
4a.	$C = -27.5 \ln I/I_{o} - 0.3$	0.3*
5a.	$C = -21.4 \ln I/I_0 + 0.8$	0.4

* Outlying points rejected in this pair of calibrations.

Based on Strip Chart Recorder Plots

1Ъ.	$C = -18.3 \ln$	$I/I_{o} + 0.8$	σ =	0.6 ppb
2Ъ.	$C = -14.6 \ln $	I/I ₀ + 1.1		0.3
ЗЪ.	$C = -20.2 \ln$	I/I ₀ + 0.2		0.2
4Ъ.	$C = -15.7 \ln $	1/1 ₀ + 1.4		0.5*
5Ъ.	$C = -11.9 \ln$	I/I ₀ + 1.9		0.4

* Outlying points rejected in this pair of calibrations.

The standard deviations include random deviations in instrumental response as well as any random deviations of the individual calibration mixtures from their reported values. It was usually possible to obtain results consistent to 0.5 ppb, but frequent calibration was required to maintain this degree of reproducibility because of day to day changes in response and drift over shorter periods of time.

7. Analysis of Departure From Beers' Law

Since calibration may be performed rather frequently, it would be advantageous in an automated system to be able to perform a calibration at a single concentration and use Beers' law to estimate intermediate concentrations. The error introduced by this procedure may be estimated by considering the relationship between the relative concentration calculated from Beers' law and the true relative concentration.

$$\frac{C_{i} - \lambda}{C_{io} - \lambda} = \frac{\ln \frac{I_{i}}{I_{o}}}{\ln \frac{I_{io}}{I_{o}}} = \left(\frac{C_{i}}{C_{io}}\right)_{\text{Beers'}}$$
(5)

where C_{10} is the initial indoor concentration of tracer and C_1 the concentration at time t, and I_{10}/I_0 and I_1/I_0 are the corresponding instrumental responses. Equation 5 assumes that there is no significant change in the parameters, a, between the measurement of C_{10} and C_1 . (C_1/C_{10}) Beers' is the relative concentration calculated from Beers' law. Equation 5 may be expressed in the form:

$$\left(\frac{C_{i}}{C_{io}}\right)_{\text{Beers}}, = \frac{\frac{C_{i}}{C_{io}} - \frac{\lambda}{C_{io}}}{1 - \frac{\lambda}{C_{io}}}$$
(5a)

To estimate the effect of errors in relative concentration on the calculated infiltration rate, the following relationship has been used.

$$\Delta \vartheta = \frac{60}{t} \left[\ln \left(\frac{C}{C_{io}} \right)_{\text{Beers'}} - \ln \frac{C}{C_{io}} \right] \text{ at time t}$$
(6)

where Δ 9 is the estimated deviation from the true infiltration rate in air changes per hour, and t is elapsed time in minutes. Plots of this relationship as a function of infiltration rate and at different values of λ/C_{io} are presented in Figures 6A and 6B. Figure 6A is based on a 1-hour measurement period and Figure 6B on 15 minutes. The error increases both with increase in λ/C_{io} and increase in infiltration rate. Also it may be shown from equations 5A and 6 that Δ 9 is reduced if the elapsed time between the measurement of the initial and fimal concentration is reduced. This is illustrated by comparing Figures 6A and 6B. From the standpoint of this error it would be desirable to make the measurement period as short as possible. However, this also increases the effect of normal random errors, particularly at low infiltration rates.

If a typical value of 15 ppb is taken as the initial concentration of tracer, for the five calibrations made with the meter, λ/C_{10} ranges from -0.03 to + 0.05. Applying Beers' law to this data, for an actual infiltration rate of one air change per hour, an average of 1.02 air changes per hour would be obtained with a standard deviation of 0.07. Using recorder data the error is larger. In this case an average from the five calibrations would be 1.15 air changes per hour with a standard deviation of 0.1. Using a 15-minute instead of a 1-hour interval the calculated rate would be reduced to 1.09 air changes per hour with a standard deviation of 0.06. As noted from Figures 6A and 6B, this error is less at lower infiltration rates, but escalates rapidly at higher infiltration rates.

The foregoing analysis of the effect of applying Beers' law to a measurement system which follows equation 4 is presented for illustrative purposes. Any system would have to be calibrated at the time of use. Also, electron capture detectors and their accompanying circuitry differ in design. Some are designed to be linear in response. However, the analysis of this particular system indicates that it is possible to obtain an approximate estimate of low infiltration rates using calibration at one concentration, but for complete reliability it would be necessary to have the capability for calibration at more than one concentration.

8. Test Operation of the Automated System

To test out the system, infiltration measurements were made in a 4-bedroom house over a 10-hour period. During the test SF_6 was introduced automatically into the furnace fan at selected time intervals, and the buildup and decay of tracer in the house was measured. Instrument response as a function of time is plotted in Figure 7. It is to be noted that a decrease in concentration appears in the figure as a rise in I/I_0 .

Two sampling methods are represented in the figure. In one method air was sampled from the main return duct just ahead of the furnace fan. In the other method blended samples were drawn from different parts of the house through a network of tubes of equal length extending to each of the rooms. These tubes converged at a sampling junction connected to a 2-cfm (57 liters/min) diaphragm pump. The automated system sampled in timed sequence from the output of this pump, from the return, and from the controlled leak secondary standard shown in Figure 4. Sampling from the return is often used in measuring infiltration rates in occupied homes. Network sampling provides an independent check on this procedure.

Infiltration rates calculated from the data in Figure 7, and corrected for non-Beers' law response of the detector, are presented in Table 1. The agreement between the two methods of sampling was within normal experimental error except when the furnace fan was not operated, as was the case during runs 2 and 4. Under these conditions it was not possible to measure the infiltration rate in the house by sampling in the return duct. Also, measurement through the sampling network showed the effects of poor mixing as evidenced by wide scatter of points. Buildings which do not have a central forced air circulation system present special problems in the measurement air leakage by the tracer dilution method.

The response of the instrument to samples from the secondary standard system is also shown in Figure 7. The general trend was a gradual decrease in I/I_0 due to drift in instrumental response with time, but the change during any 1-hour period was rather small. In addition, however, there were periods of instability which are presumably due to observed periods of greater than average wind activity.

From these tests and from subsequent measurements it is concluded that automated operation of the chromatograph itself can go for hours unattended. However, the standardization needs further development before the measurement system can be considered fully automated for unattended operation for more than an hour at a time, except for approximate estimates of infiltration rates of less than one air change per hour.

9. Acknowledgment

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10. References

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Table 1

Run No.	Network Sampling	Return Sampling
1	0.45	0.46
2	.14*	_*
3	.44	. 34
4	0*	.76*
5	.27	.23
6	.27	.27
7	.25	.25
8	.26	.26
9	.21	.22

Infiltration Rates Obtained With Experimental Automated System

* House circulating fan not operated during these periods.





Figure 2A. Picture of system for automated operation of chromatograph.

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Figure 2B. Block diagram of system for automated operation of chromagraph.



Figure 2C. Wiring diagram of system for automated operation of chromatograph.



Figure 3A. Block diagram of tracer feed system.

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FROM STEPPER 2

Figure 3B. Wiring Diagram of feed Actuating circuit.



Figure 3C. Wiring diagram of feed control circuit.



Figure 3D. Pictorial diagram of feed control system.



Figure 4. Experimental system for preparation of SF_6 dilutions in air.



Figure 5. Response of detector to measured concentrations of SF_6 .



Figure 6A. Estimated error in infiltration rate encountered when Beer's Law is substituted for actual calibrated response based on 1 hour measurement period.



Figure 6B. Estimated error in infiltration rate encountered when Beer's Law is substituted for actual calibrated response based on 15 minute measurement period.